

**REMARKS**

Claims 8-23 are pending in the application. Claims 8, 12, 15, and 18-22 have been amended. Claim 24 has been added and that claim finds basis in claim 15. Claims 9 and 10 have been canceled without prejudice or disclaimer. No new matter is involved. No further consideration or search is required. Applicants note the draft Examiner's Amendment of about the date of June 20, 2007, concerning these amendments. Regarding the amendments to claim 8, they are supported by claims 9 and 10 and track the parent patent 6,649,560. See claim 1 thereof. Consequently, no new issues or further consideration is required. Entry is requested.

Claims 8-23 have been rejected under 35 U.S.C. 112, second paragraph. Applicants respectfully submit that in view of the amendments to the claims, essentially based upon suggestions made by the Examiner, these formal matters have been overcome and should be withdrawn.

Claims 8-23 have been rejected under 35 U.S.C. 102(b) as anticipated by or in the alternative under 35 U.S.C. 103(a) as obvious over Cheminal, U.S. Patent No. 5,523,500. Applicants respectfully traverse this rejection.

Applicants respectfully point out to the Examiner that the Cheminal reference is the English-language equivalent of European Patent No. 546,883. That reference is discussed and analyzed as background art on page 2 of the present specification. Furthermore, Applicants respectfully point out that that reference has been considered and listed as references cited in parent patent 6,649,560.

As pointed out in the specification, the Chemical reference describes the preparation of bulk catalysts based on chromium and on nickel by the sol-gel method in several stages, the first consisting and forming a mixed sol of chromium III and nickel II hydroxides. This technique, which starts with a mixture of the precursors of the chromium and of the nickel, is relatively lengthy and expensive to implement. As pointed out on page 3 of the specification, Applicants have discovered that a mixed nickel-chromium catalyst which is particularly effective in the gas-phase fluorination by hydrogen fluoride of saturated or olefinic halogenated hydrocarbons can be obtained by simple impregnation of a bulk chromium oxide, with a large specific surface and with a high pore volume, with a solution of a nickel derivative. The fluorination examples commencing on page 10 of the specification illustrated in Tables I and II, in particular Examples 6-9, show simple impregnation of a commercial chromium oxide and recovery thereof. Example 10 shows the beneficial effect of nickel and Example 11 shows that a catalyst prepared with a chromium oxide which does not observe the specific surface and pore volume criteria defined in the present invention gives markedly poor fluorination results. These results teach over the reference applied in the Office Action and establish unexpected results and non-obvious subject matter.

Applicants have conducted additional experiments which illustrate the effect of the Ni/Cr ratio on the fluorination ability of the catalyst, as well as the importance of the BET specific surface and pore volume properties of the chromium (III) oxide starting materials to the fluorination abilities of the catalyst produced therefrom. The experiments and results thereof are described in the attached Declaration under 37 CFR 1.132 ("the Declaration").

The additional experiments are designated in the Declaration as "Examples 14-16". In Examples 14-16, three catalysts (Catalyst A', Catalyst A" and Catalyst E') were prepared in accordance with the procedure set forth in Example 1 of the instant specification except that the chromium (III) oxides used as starting materials had the BET specific surface and pore volume properties set forth in Table I below. The Ni/Cr ratios of the catalysts are also set forth in Table I.

**TABLE I**  
**Catalyst A', A" and E':**  
**BET Specific Surface, Pore Volume and Ni/Cr Ratio**

	<u>Catalyst</u>		
	<u>A'</u>	<u>A"</u>	<u>E'</u>
Cr (III) Oxide: BET Specific Surface (m <sup>2</sup> /g)	205	205	42.1
Cr (III) Oxide: Pore Volume (cm <sup>3</sup> /g)	0.268	0.268	0.079
Catalyst: Ni/Cr ratio	0.4	0.5	0.4

The catalysts were used for the gas-phase fluorination of perchloroethylene in accordance with the process set forth in Example 6 of the instant specification. The operating conditions and the results are set forth in Table II. The abbreviations used in Table II have the same meanings as set forth in the instant specification.

**TABLE II**  
**Examples 14-16: Fluorination Operating Conditions and Results**

	<u>Example No.</u>		
	<u>14</u>	<u>15</u>	<u>16</u>
<u>Operating Conditions</u>			
Catalyst	A'	E'	A''
Temperature (°C.)	350	350	350
Molar Ratio: HF/C <sub>2</sub> Cl <sub>4</sub>	6.89*	6.99	7.1
Molar Ratio: O <sub>2</sub> /C <sub>2</sub> Cl <sub>4</sub>	0.01	0.01	0.01
Pressure (MPa)	0.1	0.1	0.1
Contact Time (seconds)	5.0	5.4	4.8
Age of the catalyst (hrs)	24	24	30
<u>Results</u>			
Overall degree of conversion of C <sub>2</sub> Cl <sub>4</sub> (%)	62.8	53.6	54.0
Selectivity (molar %) for:			
F125	19.7	15.0	19.9
F124	35.5	38.2	32.0
F124a	2.0	1.0	1.9
F123	24.5	25.9	21.9
F123a	1.7	1.9	2.5
F122	1.7	2.1	2.5
F133a	1.6	0.9	1.3
F155	0.8	1.3	0.9
F114 + F114a	3.6	3.5	3.6
F1111	8.1	10.1	12.4
Others	0.8	0.1	1.1

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\* In the attached Declaration, the number "8" in "6.89" was handwritten in by Mr. Lacroix to correct the erroneous recitation of the number "3" that was typewritten at that location. In addition, on page 4, line 8, of the Declaration, Mr. Lacroix blanked out the second "I" in the symbol "II" to correctly indicate which Table the reported data was taken. The Declaration is submitted herewith exactly as received by the undersigned attorney. It is the undersigned attorney's understanding that Mr. Lacroix signed the Declaration after making the aforementioned corrections.

Catalysts A' and E' have the same Ni/Cr ratio but use chromium (III) oxide starting materials having different BET specific surface and pore volume properties. Catalyst A' is within the scope of the present invention, while Catalyst E' is outside the scope of the invention. As shown in Table II, Catalyst A' achieves a higher conversion rate for perchloroethylene and greater selectivity for F125 than does Catalyst E'. Thus, Catalysts A' and E' illustrate the importance of the BET specific surface and pore volume properties of the chromium (III) oxide starting materials to the fluorination abilities of the catalyst produced therefrom.

Catalysts A' and A'' have different Ni/Cr ratios but use chromium (III) oxide starting materials having the same BET specific surface and pore volume properties. Catalyst A' is within the scope of the claimed invention while Catalyst A'' is outside the scope of the claimed invention. As shown in Table II, the use of Catalyst A'' entails a significant decrease in the conversion rate of perchloroethylene, compared to the conversion rate achieved with Catalyst A'. Thus, Catalysts A and A'' demonstrate the significance of the Ni/Cr ratio to the fluorination abilities of the catalysts.

The Declaration indicates that Catalysts A and E (prepared in Example 1 and Comparative Example 5, respectively, of the instant application) both have a Ni/Cr ratio of 0.05. Thus, while it has a Ni/Cr ratio within the scope of the claimed invention, Catalyst E is outside the scope of the claimed invention because it uses a chromium (III) oxide starting material having BET specific surface and pore volume values outside the ranges recited in the claims. The fluorination properties of Catalysts A and E were tested in Example 6 and Comparative Example 11, respectively. From the results shown in Table I of the specification, it appears that at two different fluorination temperatures

(300°C and 350°C), Catalyst A provides a much greater conversion rate of perchloroethylene and a greater selectivity for F125 than does Catalyst E.

Applicants respectfully submit that the evidence set forth in the present specification, which is sworn documentary evidence, alone or in combination with the additional evidence submitted by way of declaration, obviate the rejection under 35 U.S.C. 102 and shift the burden to the Patent Office to establish anticipation.

Claims 8-23 have been further rejected under 35 U.S.C. 103 as being unpatentable over Chemical '500. For the reasons advanced above, Applicants respectfully submit that the rejection is an error and should be withdrawn.

For all the reasons advanced above, Applicants respectfully submit that the application is in condition for allowance and that action is earnestly solicited.

Respectfully submitted,

SMITH, GAMBRELL & RUSSELL, LLP

By:

Frederick F. Calvetti, Reg. 28,557

Date: November 20, 2007

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Attachment: Declaration of Eric Lacroix (5 pgs)



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Eric LACROIX, et al.

Application No.: 09/535,978

Group Art Unit : 1755

Filed: March 27, 2000

Examiner : Elizabeth D. Wood

For: BULK CATALYSTS BASED ON CHROMIUM AND ON NICKEL  
FOR THE GAS-PHASE FLUORINATION OF HALOGENATED HYDROCARBONS

DECLARATION UNDER 37 C.F.R. 1.132

Box A1

Commissioner for Patents  
Washington, D.C. 20231

Sir:

**Declaration of Eric Lacroix**

I, Eric Lacroix, declare as follows:

I am a coinventor of the subject matter claimed in the above-identified application, and am employed by the assignee of the application. My Curriculum Vitae (which sets forth, among other things, my educational background, my technical areas of expertise, the length of time for which I have been employed by the assignee, and my experience in the field of bulk catalysts) is attached hereto.

Under my direction, supervision and control, certain experiments were conducted wherein bulk catalysts based on nickel and chromium were prepared and then tested for their fluorination ability. These experiments are designated herein as "Examples 14-16" and the catalysts prepared therein are designated as Catalysts A', E' and A", respectively.

Catalysts A', E' and A" were each prepared, dried and activated in the same manner as Catalyst A was in Example 1 set forth in the above-identified application except that the chromium (III) oxides used as starting materials in Examples 14-16 had the BET specific surface and pore volume values set forth in Table I below and the catalysts had the Ni:Cr atomic ratios also set forth in Table I:

**TABLE I**  
**Catalyst A', E' and A":**  
**BET Specific Surface, Pore Volume and Ni/Cr Ratio**

	<u>Catalyst</u>		
	<u>A'</u>	<u>E'</u>	<u>A"</u>
Cr (III) Oxide: BET Specific Surface (m <sup>2</sup> /g)	205	42.1	205
Cr (III) Oxide: Pore Volume (cm <sup>3</sup> /g)	0.268	0.079	0.268
Catalyst: Ni/Cr atomic ratio	0.4	0.4	0.5

Catalysts A' and E' had the same Ni/Cr atomic ratio but used chromium (III) oxide starting materials having different BET specific surface and pore volume properties. Catalysts A' and A" had different Ni/Cr ratios but used chromium (III) oxide starting materials having the same BET specific surface and pore volume properties. Catalyst A' is within the scope of the claims of the instant application. Catalysts E' and A" are outside the scope of the instant claims.

In Examples 14-16, Catalysts A', A" and E' were then used in the gas-phase fluorination of perchloroethylene under the operating conditions set forth in Table II below. The results are also set forth in Table II. The abbreviations used in Table II have the same meanings as the terms used in Tables I and II set forth in the instant application.

**TABLE II**  
**Examples 14-16: Fluorination Operating Conditions and Results**

	<u>Example No.</u>		
	<u>14</u>	<u>15</u>	<u>16</u>
<u>Operating Conditions</u>			
Catalyst	A'	E'	A"
Temperature (°C.)	350	350	350
Molar Ratio: III/C <sub>2</sub> Cl <sub>4</sub>	6.89	6.99	7.1
Molar Ratio: O <sub>2</sub> /C <sub>2</sub> Cl <sub>4</sub>	0.01	0.01	0.01
Pressure (MPa)	0.1	0.1	0.1
Contact Time (seconds)	5.0	5.4	4.8
Age of the catalyst (hrs)	24	24	30



Results

Overall degree of conversion of $C_2Cl_4$ (%)	62.8	53.6	54.0
Selectivity (molar %) for:			
F125	19.7	15.0	19.9
F124	35.5	38.2	32.0
F124a	2.0	1.0	1.9
F123	24.5	25.9	21.9
F123a	1.7	1.9	2.5
F122	1.7	2.1	2.5
F133a	1.6	0.9	1.3
F155	0.8	1.3	0.9
F114 + F114a	3.6	3.5	3.6
F1111	8.1	10.1	12.4
Others	0.8	0.1	1.1

As shown in Table II, Catalyst A' achieved a higher conversion rate for perchloroethylene and greater selectivity for F125 than did Catalyst E'. As noted above, Catalysts A' and E' had the same Ni/Cr atomic ratio but used chromium (III) oxide starting materials having different BET specific surface and pore volume values. The BET specific surface and pore volume values of the chromium (III) oxide used in the preparation of Catalyst A' were both within the ranges of these values recited in the claims of the instant application. The BET specific surface and pore volume values of the chromium (III) oxide used in the preparation of Catalyst E' were both outside the ranges of these values recited in the claims of the instant application.

As further shown in Table II, the use of Catalyst A" entailed a significant decrease in the conversion rate of perchloroethylene, compared to the conversion rate achieved with Catalyst A'. As noted above, Catalysts A' and A" had different Ni/Cr atomic ratios but used chromium (III) oxide starting materials having the same BET specific surface and pore volume values. Catalyst A' had a Ni:Cr atomic ratio (i.e., 0.4) within the range recited in instant claim 1 of the present application (i.e., 0.02-0.4), whereas Catalyst A" had a Ni:Cr atomic ratio (0.5) outside the range recited in claim 1.

The Examples, including the Comparative Examples, set forth in the instant application were also conducted under my direction, supervision and control.

Although not stated in the instant specification, the catalysts prepared in Example 1 and Comparative Example 5, i.e., Catalysts A and E, both had a Ni/Cr ratio of 0.05. As is described in the specification, Catalysts A and E differed in the BET specific surface and pore volume values of the chromium (III) oxide used as a starting material. Catalyst A was within the scope of the instant claims, whereas Catalyst E was outside the scope of the instant claims. As further indicated in the specification at Table I: therein, the fluorination ability of Catalyst A was superior to that of Catalyst E.

I declare that all statements made herein of my own knowledge are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

Respectfully Submitted,

February 6, 2002  
Date

Eric Lacroix  
Eric Lacroix

## **Eric Lacroix Curriculum Vitae**

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69480 Ambérieux d'Azergues  
FRANCE

### **Educational background :**

Graduated from the « Ecole Nationale Supérieure de Chimie de Strasbourg » (Strasbourg University, France) in June 1984

September 1985 – september 1988 : PhD in organic synthesis

### **Professional background :**

Employed by Elf Atochem (now called ATOFINA) since September 1985

September 1988 to October 1996 : scientist in the Department for Fluorinated Derivatives of the Lyon Research Center of Elf Atochem (called CRRA)

Since October 1996 : Head of the Department for Fluorinated Derivatives

Since January 1999 : Head of the Department for Catalysis

Since I have been working for Atofina, I am making research on CFCs (chlorofluorocarbures) substitutes. I am in charge of research linked to industrial process development of fluorinated compounds, notably catalysts for fluorination reactions. With Atofina, I have filed many patents based on bulk or supported catalysts.